5.5 Inductively Coupled Plasma-Mass Spectrometry for Analysis of Microliter Samples and Solids

Technology Need

This project will provide analytical technology needed to support a wide variety of remediation problems, such as: (1) real-time measurement of transuranic elements and other radionuclides, (2) rapid measurement of RCRA metals in a wide variety of aqueous or organic solutions, and (3) removing the general need for sample dissolution for accurate quantitative determination of metals. The overall cost of such analyses will also be reduced because the amount of radioactive waste samples will be reduced to microliter quantities or less, which greatly simplifies containment concerns.

Technology Description

Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) is already a highly sensitive and selective method for elemental and isotopic analysis. This project will investigate the ability of a microscale nebulizer called a monodisperse dried microparticulate injector (MDMI), to improve the sensitivity, speed, accuracy, and precision of ICP-MS for determination of stable elements and radionuclides.

Essentially, a micropump creates uniform wet droplets that are dried carefully and then introduced into the plasma for conversion into atomic ions. There is little or no waste solution; 100% of the sample reaches the plasma. Exposure to radioactivity and waste cleanup problems during analysis will also be greatly reduced because the nebulizer requires only nanoliter to microliter volumes of solution.

Specifically, two related projects are under study: (1) direct analysis of very small solution volumes, and (2) on-line calibration for laser ablation ICP-MS, so that solids can be analyzed directly with better accuracy than is now achievable.

The analytical capabilities of MDMI-ICP-MS, such as detection limits and tolerance to concentrated sample matrices, will be evaluated thoroughly for real samples of interest in waste remediation. This sample introduction technology is potentially applicable to existing ICP-MS devices used for analyses that support waste clean-up. It should also be suitable for field use with a mobile ICP-MS device in a van.

Benefits

The major benefits are more sensitive measurement of radionuclides and stable elements with little waste solution, and direct analysis of solids with simple, accurate calibration procedures that do not require matrix-matched standards.

Collaboration/Technology Transfer

The MDMI is an advanced prototype donated by Perkin-Elmer SCIEX. They intend to offer the MDMI as a commercial product and are eager to collaborate with us to evaluate its suitability for these special applications. The Principal Investigator has been associated with SCIEX since the early days of ICP-MS (~1982).

Accomplishments

This project began in FY95. The main accomplishments to date are the following:

- Detection limits are approximately 0.1 fg of uranium in a solution volume of 0.1 nL. This represents 250,000 atoms of uranium. Detection limits are similar for most other elements
- Improvements in precision from ~2% to 0.1% relative standard deviation for measurement of ion ratios in solids by laser ablation ICP-MS
- Fundamental characterization of matrix interferences and substantial operational improvements in the reliability and consistency of MDMI-ICP-MS

Initial feasibility experiments on calibration of laser ablation ICP-MS with solution aerosols are underway.

TTP Information

Inductively Coupled Plasma-Mass Spectrometry for Analysis of Microliter Samples and Solids technology development activities are funded under the following Technical Task Plan (TTP):

TTP No. CH15C241 "Inductively Coupled Plasma-Mass Spectrometry for Analysis of Microliter Samples and Solids"

Contacts

R. S. Houk
Principal Investigator
Ames Laboratory
Iowa State University
108 Spedding Hall
Ames, IA 50011
(515) 294-9462

Richard Baker
Technical Program Officer
USDOE-Chicago Operations
9800 S. Cass Ave
Argonne, IL 60439
(708) 252-2647

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